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AGRICULTURAL UTILIZATION OF MAOMING OIL SHALE ASH

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Mineral matter accounts for 60-80 % of oil shale, so a tremendous amount of solid waste during pyrolysis or burning of oil shale is discarded, occupying land and causing pollution to the environment. The oil shale pyrolysis plants in Maoming Petrochemical Company in China have been operated for 30 odd years and cumulatively more than 200 million tons of shale ash covers a dumpyard of 7 sq. km. The leached water already causes direct damage to farmland and fishpond.

The present study deals with samples from different depths of the dumpyard and makes a comprehensive analysis of the elements and matter useful and harmful to plants and crops. Experiments show that high potassium content in Maoming shale ash is good for plant growth and the cadmium content at some sampling points is slightly higher than the national standard. Field tests of growing peanuts and soybean succeeded with allowable heavy metal and benzopyrene contents in the edible parts.

Agricultural use of shale ash improves environmental quality and makes use of wasteland being economically promising.

Introduction

Shale oil production from oil shale pyrolysis had been in operation for more than 60 years in China [1]. The disposal of solid waste from pyrolysis plants not only occupies land but also causes pollution to atmosphere, water and farmland during dusting and drainage. Part of shale ash is used for backfill of mines or manufacture of building materials, but the pile up of shale ash has caused damage to the environment in the surroundings.

In the sixties 40 sets of pyrolysis retorts 200 tons per day of oil shale each were built and operated in Maoming Petrochemical Company [1]. Piling up of shale ash over more than 30 years has occupied 7 sq. km of dumpyard 20-30 m above surface with a total amount of 240 million tons. The aim of the present work is to study agricultural utilization of Maoming shale ash with experimental and field tests of plants and crops cultivation on shale ash.

Prior Work on Shale Ash Utilization

Besides backfill of mines and manufacture of cement, the agricultural utilization of shale ash, such as plant cultivation and fertilizer application have been tested in the US, Estonia, Russia and Australia.

The Green River shale ash in the US generally has a higher pH and higher content of calcium, potassium and various salts, while lacks bio-available nitrogen and phosphorous. The research work conducted by Schmehl and McCaslin showed that good growth of wheat grass, cultivated on the shale ash was obtained, if the ash was pre-treated with water to remove excess salts and both N and P fertilizers were applied [2].

Heistand and Limbach conducted the research work on the natural invasion of native plants on retorted oil shale. The concentrations of trace elements (Mo, F, Cu and B) did not show any large differences in new growth from saltbush and fireweed plants whether growing on retorted shale or native soil [3].

Stark and Redente have made analysis of the concentration of As, B, Cu, Mo, Se present in several plants cultivated on leached shale ash, the results showed that grass family plants may be used as domestic animal feed [4, 5]. Barnhisel has conducted the research work on revegetation of Eastern US spent oil shale for more than ten years. He has made investigation on the chemical composition of plant issues growing on spent shale, such as Ca, Mg, K, P, Na, Mn, Fe, etc. and their influence on the tall fescue yield, and concluded that Cu and Ni levels are within the acceptable range for animals [6, 7].

In Estonia, where the soil is acidic, kukersite oil shale and shale ash containing alkaline materials and nutritional elements have been used as fertilizer and soil ameliorator since 1944. Pets and Vaganov analyzed and classified 52 macro- and microelements of shale ash from Baltic Power Plant [8, 9]. They made a detail investigation on the concentrations of 20 elements (including Fe, Co, Ag, Au, As, Sb, Ni, Rb, Cs, Ba, Se, La, Sm, Tb, U, Th, Zr, Hf, Th, and Ce) in species of meadow grass grown on the control lots and on the lots after chalking of soil by shale ash. The share of Ba, As, Cr, Br, Fe, Co, and Ni in nutrition of plants has been established [10]. Turbas pointed out that Estonian oil shale ash obtained from thermal power plants could be successfully used as a lime fertilizer with great capacity for neutralization. It contains, in addition to calcium compounds, considerable amounts of potassium, magnesium and sulfur as well as small quantities of other elements needed for plant nutrition [11, 12].

In Australia, Marshall made the assessment of revegetation experiments at Rundle. He showed that when spent shale was added to either claystone or overburden good seedbed conditions were created [13, 14]. Tait made analysis of concentrations of 13 trace metals and cations (including Na, K, Ca, Mg, Fe, Al, Cu, Zn, Cd, Cr, Ni, Pb, and As) in topsoil overburden, weathered Rundle oil shale, and spent shale, as well as in pasture grass growing on shale and ash. The results indicated that shale ash does not increase the availability of any of the metals to the grass. It was also shown that the concentrations of Cu, Zn, Ni and As, occurring in gayana grass growing on spent shale, are all well below documented levels which produce toxic effects or yield reductions [15].

In China, agricultural environmental protection station in Maoming preliminarily conducted the research work on the investigation of the planting paddy on the oil shale ash and the effect of shale ash on the improvement of soil quality.

However, the composition of oil shale and its ash change significantly in different countries and places. Generally speaking, oil shale in the US and Estonia contains higher content of calcium oxide and magnesium oxide, lower content of aluminum oxide and silicon oxide, and more carbonates in inorganics. This oil shale belongs to calcareous shale with water-hardening ash. Maoming oil shale in China has a higher content of aluminum oxide and silicon oxide, lower content of calcium oxide and magnesium oxide. This oil shale belongs to clayey shale with non water-hardening ash. The microelements containing in shale ashes are also different from each other. Therefore, utilization of shale ash should be based on its specific conditions and begin with a systematic study of its properties.

Methods of Sampling and Analysis

Sample Collection

Shale ash samples were collected at typical sites in the waste disposal area. Adjacent *in situ* soil samples were also collected for comparison.

Plants cultivated on shale ash pile were collected in harvest season. Soils derived from shale ash in the cultivated plots were collected simultaneously.

Sample Preparation

Soil and ash samples were air-dried, ground and sieved to pass through 60 or 100 mesh according to the requirement of specific analytical item.

Plant samples were washed using deionized water and dried under low temperature. Then the samples were cut and ground.

Chemical Analysis

Shale ash and soil samples were analyzed to determine the concentrations of a series of elements with the application of the following procedures:

Determination of Mineral Elements

Samples were melted under 900 °C using Na_2CO_3 as the flux. Hydrogen chloride was added to dissolve the cooled sample. When Si was moved, the contents of Ca, Mg, Fe, Al were determined with Inductively Coupled Plasma Photospectrometer (ICP 9000 SP).

Determination of Total Concentration of Nutritional Elements - N, P, K

Total concentration of **nitrogen** was determined by using Kjeldahl method. Samples were heated with concentrated H_2SO_4 so that nitrogen in the form of organic compound was converted into inorganic form, forming $(NH_4)_2SO_4$. Then, with the addition of concentrated NaOH, NH_3 was distilled and absorbed by boric acid solution and determined by titration.

Total concentration of **phosphorus** was determined with colorimetric method. Samples were heated with concentrated H_2SO_4-$HClO_4$. Then the contents of P were determined by using $(NH_4)_2MoO_4$-$KSbOC_4H_4O_6$ colorimetric method.

Total concentration of **potassium** was determined by digesting the samples with the addition of HF-$HClO_4$. After dissolving the matter with hydrogen chloride, potassium was determined with the application of flame photometer.

Determination of Available Nutritional Elements - N, P, K

Alkali decomposable **nitrogen**: Through the hydrolysis process of the samples under alkali condition, part of the nitrogen was converted to form NH_3. NH_3 spread in a container and was absorbed by H_3BO_3. Then NH_3 was determined by titration and the concentration of available nitrogen was calculated.

Available **phosphorus**: For acidic or neutral samples, phosphorus was extracted by 0.03 mol/l NH_4F-0.025 mol/l HCl; for alkali samples, phosphorus was extracted by 0.5 mol/l $NaHCO_3$. Extractable P was determined by $(NH_4)_2MoO_4$-$KSbOC_4H_4O_6$ colorimetric method.

Available **potassium**: Potassium was extracted by using 1 mol/l NH_4OAC (pH 7.0) and determined by using ICP.

Determination of the Concentrations of Heavy Metals and Other Microelements

Heavy metals: Samples were put into pressurized vessel, heated and digested with the addition of HNO_3-$HClO_4$-HF. Then diluted HNO_3 was applied and the digested liquid was analyzed using atomic absorption spectrophotometer (Hitach 18080).

Metalloids: For the determination of arsenic, samples were digested with concentrated HNO_3-H_2SO_4 and the concentration of As was measured with ultraviolet spectrophotometer. As for selenium, samples were treated in HCl solution, potassium hydroboron used as reducing agent. Se in samples was converted to the form of volatile gas and the concentration was determined with atomic absorption spectrophotometer using Ar as carrier gas.

Germanium: Samples were digested under pressure with HNO_3 - HF and determined by using ICP/MS.

Radioactivity Measurement

Weighed samples were put into the special box, then sealed for 3-4 weeks and determined by ADCAM 1 spectrometer (ORTEC).

For Plant Samples

Macroelements (Fe, Al, Ca, Mg, etc.) and Heavy Metals Determination

Samples were heated and digested with HNO_3-$HClO_4$, and analyzed by using ICP or atomic absorption spectrophotometer.

Radioactivity Measurement

The plant samples were put into cylindrical plastic box and pressed by two pieces of organic glass plates. Then sealed with wax, after 3 weeks, the radioactivity was measured by ADCAM 1 spectrometer.

Benzopyrene Determination

Benzopyrene in samples was extracted by organic solvent. The extracts were purified by chromatographic column. When concentrated, benzopyrene content was determined by high-pressure liquid chromatography.

Fruit Quality Determination

Vitamin C was analyzed by titration using 2,6 dichloro-phenolic agent.

Reducing saccharides: After removal of protein samples were heated and titrated by alkaline cupric tartarate.

Agricultural Utilization of Maoming Shale Ash

Natural Environment

Maoming is located in the southwestern part of Guangdong Province in south China. The subtropical monsoon climate there provides sufficient illumination and strong radiation, with a high annual average temperature of 23 °C, and long summer time of more than half a year. The whole year is frost-free. Rainfall is plentiful with a long rainy season and average yearly precipitation 1705 mm. Rainfall occurs mostly in April to September, accounting for 84 % of the whole year. Such a climate is very favorable to agriculture and forestry. The shale ash dumpyard in Maoming is not far from the shale oil production plants. Dark colored shale ash absorbs heat readily and is susceptible to spontaneous combustion in the open air. The exposed shale ash at noon in summer can reach 40 °C on the surface, 2-3 °C higher than the surrounding. Besides, the pollutants leached out by rain cause contamination to the environment. The leached water from dumpyard is strongly acidic with pH value 4-5. The cadmium content reaches 0.015 mg/l, higher than the allowable surface water standards. The pollutants are harmful to crops and fish, threatening human health.

Table 1. **Composition of Organic and Mineral Matter (dry basis, %)**

Organic matter				
C	H	N	S	O
17.47	2.83	0.59	1.25	6.12
Mineral matter				
SiO_2	Al_2O_3	Fe_2O_3	CaO, MgO	Na_2O, K_2O
62	24	10	2	2

Chemical Composition of Shale Ash

Composition of Maoming oil shale is presented in Table 1.

Mineral matter accounts for 60-80 % of Maoming oil shale and consists of quartz, kaolinite, mica, corundum, dolomite, gypsum and pyrite.

Grain Size and Elemental Composition of Shale Ash

Different types of dumped shale ash were observed in northern and southern piles in the dumpyard: black shale ash from pyrolysis retorts, red shale ash from burning, and brown unused oil shale fragments. Grain size distribution is shown in Table 2.

The grain size of shale ash is larger than soil in general, and consequently larger pore space is good for aeration but at the same time easy to lose water. Pyrolysis shale ash has 50-60 % of coarser grain of 0.5-0.3 mm, while shale ash from burning has rather uniform grain size with 21 % of that fraction.

Table 2. **Distribution of Shale Ash by Grain Size (%)**

Type of ash	Grain size, mm				
	>0.5	0.5-0.3	0.3-0.125	0.12-50.105	<0.105
Black ash:					
In northern pile	3.8	53.44	37.25	1.6	1.19
In southern pile	4.81	62.34	26.86	0.85	0.92
Red shale ash	14.36	21.63	57.75	2.06	1.45

Metallic elements in shale ash can be either favorable or harmful to plant growth. Typical samples from different locations in the dumpyard were collected for macroelement nutrients, macroelement mineral matter and microelement analysis and comparison with national standards. The contents of main nutrients, heavy metals and microelements are shown in Tables 3 and 4.

Evidently, compared with black ash, with further combustion and oxidation, red ash contains significantly less nitrogen, and a higher level of bio-available potassium. It is good for crops, but its lower content of phosphorus needs its supplementary supply and effective utilization in agriculture and forestry application.

Table 3. **Main Nutrients**

Type of ash	N, %	P, %	K, %	Bio-available K, mg/kg
Black ash	0.259-0.540	0.035-0.084	1.40-2.23	148-162
Red ash	0.030-0.050	2.00-2.38	—	178-370

Table 4. **Microelement Content in Piled Shale Ash (mg/kg)**

Element	Content in shale ash	National standard		Medium value of soil in Guandong	Upper limit of soil in Guangdong
		Orchard	Farmland		
Cu	30.71-40.51	150	50	11.4	77.1
Zn	55.67-154.4	200	—	—	158.6
Cr	57.60-62.06	150	—	34.8	178.6
Mn	299.16-349.56	—	—	151	1277
Mo	3.64-14.42	—	—	7.0	18.3
Ni	27.08-27.63	—	40	8.8	53.9
Co	12.71-31.11	—	—	5.0	22.93
Pb	31.21-49.88	—	250	28.9	106.4
Cd	0.130-0.404	—	0.30	0.04	0.20
As	6.82-22.42	—	40	7.1	28.6
Se	0.1-0.68	—	—	0.233	0.792
B	15.13-18.05	—	—	10.1	99.2
Ge	2.4-2.5	—	—	1.6	2.42

Based on the elemental composition of shale ash, as shown in Table 4 and compared with the standard, its possible biological effect can be detailed as follows.

(1) Cu, Zn, Cr, Mn, and Mo. These elements are essential in plant growth but their excessive content may cause a poisoning effect. However, as shown in Table 4, their contents are below the upper limit in Guangdong soil, and the content of Cu, Zn, Cr in shale ash is moderate and does not exceed national standards.

(2) Pb, and Cd. These elements are not needed in plant growth. Their high content will cause a poisoning effect [11]. The Pb content in shale ash is much lower than the national environment quality standard for the soil (see Table 4). The Cd content in samples from some sampling points is slightly higher than the standard, so careful monitoring is required in application and specific measures are to be taken. Forestland can tolerate a higher Cd content than farmland and orchard, so trees of economic value can grow in locations with a higher Cd content.

(3) Ni, and Co. They are not the elements required for plants and are necessary only for some specific plants. Poisoning may occur if the content is too high. The average Ni and Co content in shale ash is within the permissible range (see Table 4).

(4) As, Se, B, and Ge.

As could stimulate growth at low concentrations, but could inhibit it at high concentrations. The As content in shale ash is safe (see Table 4).

Se favors plant growth at low concentrations, but not at overly high concentrations. Se is needed for human and animals [11]. The Se content in shale ash is above the medium value but below the upper limit in the soil in Guangdong Province, but the national standard for Se content in soil is lacking.

B is an element necessary for plant growth. Deficiency of boron in soil will cause plant diseases, but too much boron is also harmful. The boron content in Maoming shale ash is much lower than the upper limit in Guangding soil.

Ge. Recent studies show that germanium is good at appropriate concentration but harmful at higher concentrations as an element of low toxicity. The Ge content in shale ash is equivalent with the upper limit in local soil.

The levels of **radioactive elements** (U, Th and Ra) are listed in Table 5. Their content is basically within the normal range in natural soil. Black shale ash accounts for the largest proportion in the dumped ash, so the radiation in dumpyard does not exceed the allowable level.

Table 5. **Determination of U, Th, Ra and Benzopyrene in Shale Ash**

Type of ash	U, μg/g	Th, μg/g	Ra, Bq/g	Benzopyrene, μg/kg
Black shale ash in southern pile	7.55	31.7	0.104	18
Red shale ash in southern pile	11.90	36.2	0.118	<0.1
Black shale ash in northern pile	7.86	29.9	0.142	9.3
Unweathered shale ash from burning	8.31	37.6	0.083	1.5
Soil in Guangding (95 %)	12.98	73.90	56.33	<0.1

Benzopyrene is a strongly carcinogen condensed-ring aromatic hydrocarbon, which is representative of polynuclear aromatic hydrocarbons. The benzopyrene content in different types of shale ash is also shown in Table 5.

It is suggested that a background concentration plus 20 μg/kg for the benzopyrene could be considered the permissible level. The benzopyrene content in Maoming ash is about 0.1-18 μg/kg. The maximum value is within the safe range [16].

Crop Cultivation in Shale Ash

Taking into account the elemental analysis of the shale ash, four types of fertilizer application were designed for agricultural utilization (Table 6).

The best design for dumpyard test was selected by orthogonal experiments in climatic simulator, simulating the climate temperature and humidity of Maoming. Further analysis was made with the crop and plants cultivated *in situ* field dumpyard on shale ash pile according to the 4 types of treatment and also on red soil as comparison, thus harvested in July 1996 (Tables 7-9).

The greatest concern is benzopyrene and cadmium. From the above analysis we can summarize that:

(1) Benzopyrene: The benzopyrene content in peanut, soybean and vegetables is lower than the national standard, taking the standard for wheat and rice as reference.

(2) Cadmium: For peanut, below the GB standard for tuberous, for soybean, below the standard for rice.

Table 6. **Types of Shale Ash Application (kg)**

Treatment	Shale ash	Calcium superphosphate	$(NH_4)_2SO_4$
1	200	25	4
2	200	50	8
3	400	25	8
4	400	50	4

Table 7. **Elemental Analysis of Peanut and Soybean (mg/kg)**

Treatment	Cd	Pb	Zn	Se
Peanut				
Control*	0.074	0.224	37.13	0.033
1	0.032	—	32.45	0.027
2	0.052	0.190	31.22	—
3	0.087	0.140	32.45	—
4	0.052	0.141	34.18	0.039
Soybean				
Control	0.136	0.871	34.04	0.029
1	0.113	0.763	37.95	0.065
2	0.047	0.599	35.91	—
3	0.030	0.480	36.21	—
4	0.036	0.510	38.41	0.027
National standard				
Rice	<0.2			
Tuberous	<0.	<1 (Soy product)	<100 (Soy product)	<0.3 (Soy product)
	GB1238-84	GB11671-89	GB13106-91	GB13105-91

*Local red soil as control.

Table 8. **Determination of Cd, Pb, Zn, Se in Vegetables (mg/kg)**

Treatment	Cd	Pb	Zn	Se
A leafy vegetable				
3	0.0002	0.0069	0.462	0.0006
4	0.0003	0.0058	1.262	–
Cowpea				
1	0.0008	0.0074	0.387	0.0007
3	0.0011	0.0030	0.528	–
Hairy gourd				
4	0.0017	0.0342	1.019	0.0008
Green gourd				
4	0.0010	0.0215	1.183	0.0008
National standard				
	<0.05	<1.0	<20	<0.1
	GB11238-84	GB11671-89	GB13106-91	GB13105-91

Table 9. **Content of Benzopyrene**

Crop	Treatment	Benzopyrene, μg/kg
Peanut	Control	0.120
Peanut	*3*	0.074
Soybean	*4*	0.270
Cowpea	*1*	0.055
Hairy gourd	*4*	0.006
Melon	*4*	0.016
Towel gourd	*4*	0.022
National standard GB7104-94		<5 for rice, wheat

(3) Lead: Lower than national standard.

(4) Zinc: Soybean and peanut are rich in zinc, as an essential element (lower than standard).

(5) Selenium: Fairly rich, meeting the requirement of food hygiene.

(6) Cd, Pb, Zn, and Se in vegetables all met the GB standard as shown in Table 8.

Besides, the mercury content analyzed is also below the limitation:

Peanut (treatment [3]): 8.3×10^{-4} mg/kg
Soybean (treatment [4]): 9.0×10^{-4} mg/kg
National standard GB2762-81 <0.02 mg/kg (grain)
<0.01 mg/kg (potato)

Prospect of Crop Cultivation on Shale Ash Dumpyard

Crop cultivation on Maoming shale ash dumpyard has the following advantages.

- Fairly abundant potassium in shale ash, and high level of effective potassium favour the growth of crops.
- Radioactivity of shale ash is within the safe range. Most of the heavy metals and metalloids of chromium, lead, arsenic meet the environment standard of soil.
- The content of benzopyrene and heavy metals in vegetables meets the requirement of food hygiene. The content of cadmium and lead meets the limitation, soybean and peanut are rich in zinc and selenium.
- The disadvantage is low content of calcium and magnesium oxides, strong acidity and low pH value, low phosphorus content. The content of cadmium, which is non-essential for living organisms, in shale ash varies around 0.3 mg/kg (national standard).

Summary

In summary, agricultural utilization of discarded shale ash has been studied in terms of its advantages and disadvantages through a systematic analysis and field cultivation testing. Macro- and microelements and metals occurring in crops, plants and shale ash have been analyzed, the contents meet the national standard or upper limit of Guangdong soil. Crop cultivation on shale ash dumpyard is feasible and promises economic benefits as well as improvement of environment.

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